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The Transference Number, Conductance, & Ionization of Hydriodic Acid

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THE TRANSFERENCE NUMBER, CONDUCTANCE, AND IONIZATION OF HYDRIODIC ACID

BY

VEE GIH CHU

THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

CHEMICAL ENGINEERING

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

VEE GIH CHU

ENTITLED THE TRANSFERENCE NUMBER, CONDUCTANCE, AND IONIZATION OF HYDRIODIC ACID

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF SCIENCE IN CHEMICAL ENGINEERING.

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THE TRANSFERENCE NUMBER, CONDUCTANCE, AND IONIZATION OF HYDRIODIC ACID.

I. Purpose of the Investigation.

The very careful measurements by A. A. Noyes and his associates[1] of the conductance and transference number of hydrochloric acid and of nitric acid indicate that the hydrogen ion has an equivalent conductance of 365 at 25° in all solutions above 0.02N. concentration. On the other hand, the value, 350, is obtained for the equivalent conductance of the hydrogen ion by extrapolation of conductance data of acids to zero concentration. Thus, the degree of dissociation of strong acids, calculated by means of the conductance ratio, $\frac{\Lambda_c}{\Lambda_0}$, can differ by 3 or 4 per cent, depending on whether the conductance of the completely dissociated acid is assumed to be equal to its conductance at zero concentration, or to the sum of the equivalent conductances of the ions at the concentration under consideration. Since the latter assumption appears to the writer to be a more reasonable one, it is his purpose in this investigation to determine the degree of dissociation of hydriodic acid without using the value of the conductance of zero concentration. In order to do this it is necessary to know the transference number of the acid and its conductance at various concentra-

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1) Noyes and Sammet, J Am. Chem. Soc. 24, 944 (1902)
Noyes and Kato, J. Am. Chem Soc. 30, 318 (1908)

tions. Moreover, it is desirable to take into consideration the effect of viscosity on the conductance of the stronger solutions. The measurements of the transference number, conductance, and viscosity of hydriodic acid, will be described first and later, the calculation of the degree of dissociation.

II. Transference Number Measurement.

The transference number was determined by the Hittorf method with the use of a silver anode and a silver iodide cathode. At the end of each experiment three middle portions as well as both electrode portions were analyzed in order to make sure that no mixing had occurred during the progress of the experiment.

Apparatus. - The apparatus employed is shown in the accompanying diagram (Fig 1). It was designed to reduce the danger of conviction currents due to local heating during the passage of the current, which, of course, would mix the various portions of the solution and vitiate the results. In order to accomplish this the constricted portions of the apparatus were, with one exception, placed at the top so that any heating, which would make the solution less dense, would not cause stirring. The exception occurs is the stop-cock at the bottom of the anode section, A. This was made small on account of the difficulty in constructing one of large bore as the rest of the apparatus. Convection currents at this point, however, could only cause stirring within the anode portion and so would result in no harm. Moreover, the various sections of the apparatus were made of such a size that the samples withdrawn for analyses would amount to about 250 cc. each, thus making it possible

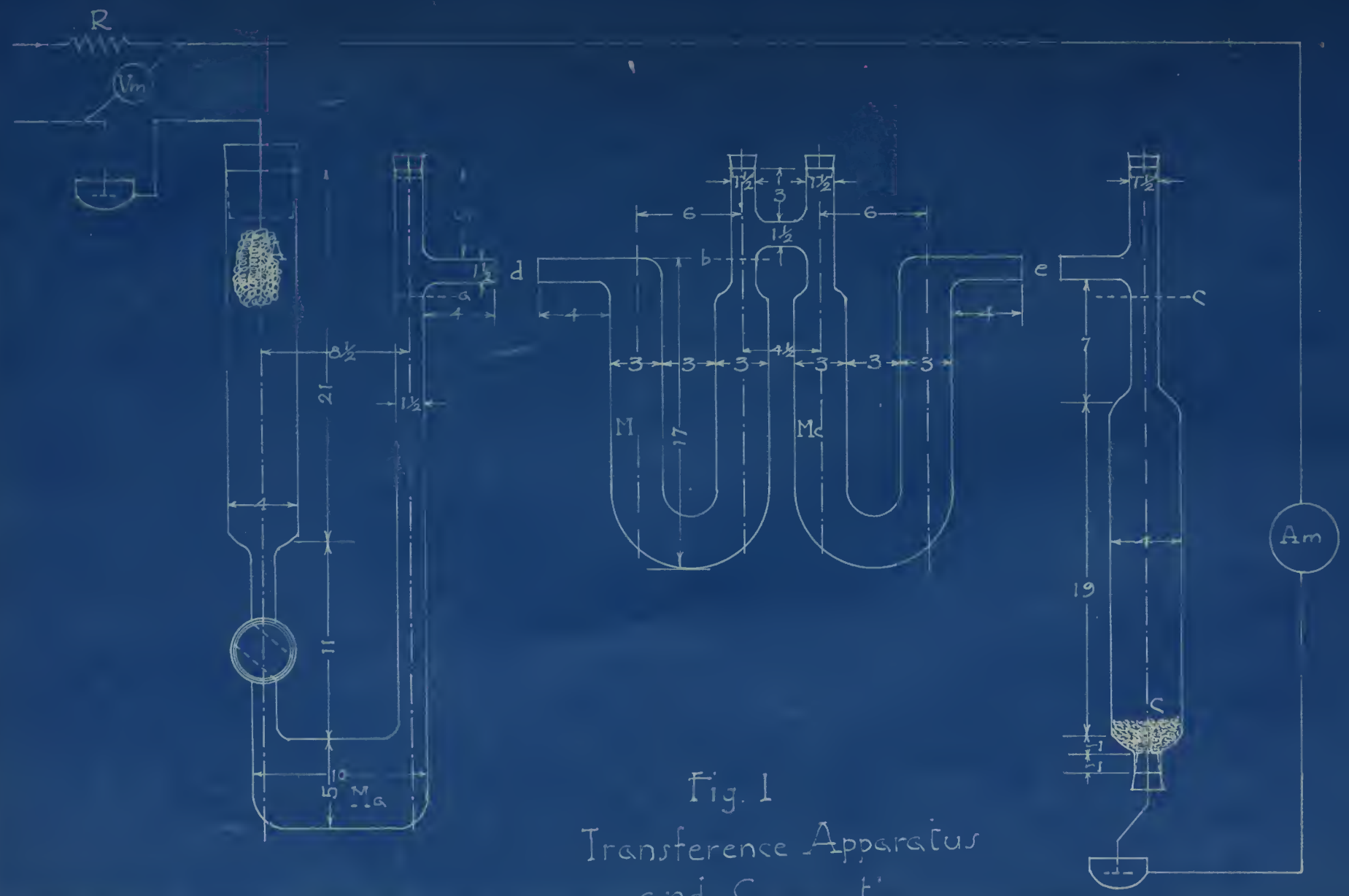

Fig. 1
Transference Apparatus
and Connectios

to secure an accuracy of 0.01 per cent in the analyses of even quite dilute solutions. The apparatus was made in three sections which were connected by rubber tubing at the points d and e. From the anode, A, to the stop-cock was designated the anode portion, and from the cathode, C, to the dotted line c, the cathode portion. The remainder of the apparatus was designed to contain the three middle portions which were called the anode middle, the middle, and the cathode middle portions. These are indicated in the diagram by M_a, M, and M_c, respectively.

The anode consisted of a silver spiral of several hundred turns of fine wire in order that it might have a large surface in contact with the solution. Before using it was dipped in warm nitric acid until it was attacked violently, then washed with water several times. After being dried, it was hung up near the top of the anode section, and connected to the lead wire of the circuit by a silver wire.

The cathode consisted of a few turns of silver wire, covered with an excess of silver iodide which had been prepared by precipitating a hot 0.03 normal solution of silver nitrate with 0.03 normal potassium iodide. Before use, the silver wire was cleaned in warm nitric acid and coated with a thin layer of silver iodide produced by using the silver wire as anode in the electrolysis of a solution of potassium iodide.

The coulometers used in this investigation were of the ordinary type, the cathode consisting of a platinum dish and the anode of a silver button wrapped in a filter paper. The anode was made of electrolytic silver. Two coulometers were used,

one connected to the anode and the other to the cathode as shown in Fig. 1, in order to detect any loss of the current during the experiment.

Preparation of Materials. - All the water used was conductivity water prepared in a special still.

The hydriodic acid was prepared in the following manner. Crude iodine was dissolved in a solution of potassium iodide and distilled. The distilled iodine was dissolved in water, and reduced by pure yellow phosphorus on the steam bath. The crude hydriodic acid so obtained was distilled twice, the fraction boiling just above 100° C. being rejected as it was chiefly water. The solution obtained by the second distillation boiled at 127°C. and consisted of the constant boiling hydriodic acid. This was then diluted to about the desired concentration and shaken a number of times with pure benzene (thiophen free) to remove the free iodine which was continually liberated, especially when the solution was exposed to light. In this way a practically colorless solution was obtained. Before use a sample of the above solution was titrated with a standard sodium carbonate solution, so that its approximate strength was determined. The stock solution was then diluted exactly to the desired concentration. This solution of hydriodic acid was always prepared and decolorized immediately before using. On analysis it was found to contain less than 0.01 per cent of free iodine, and there was no noticeable increase during the transference experiment.

The silver nitrate used both for analyses and in the coulo-

meters was Kahlbaum's c. p. silver nitrate. A 20 per cent solution was used in the coulometers, this being the concentration employed by Guthe in his work at the United States Bureau of Standards.[1] It was always freshly prepared and never used more than twice. A 0.03 normal silver nitrate solution was used in the analyses.

Experimental Proceedure. - Before a run the apparatus was thoroughly cleaned with warm chromic acid solution, and after washing with water and drying with alcohol, the three parts of the apparatus were placed in their supports and connected at d and e. The stop-cock was slightly greased with vaseline, the surface immediately opposite the cross tube being, however, left free from vaseline so that on closing the stop-cock the lubricant would not come in contact with the solution in the apparatus. After the insertion and alignment of the stop-cock, a thin layer of melted beeswax was applied to the joint between the key and the sheath, both front and rear, to the joints of rubber tubing at d and e and at the bottom of the cathode, also to the joints of stoppers at all parts. This beeswax seal entirely prevented all connections between the solution in the apparatus and the water in the thermostat in which the apparatus was to be placed.

After the insertion of the electrodes, the cathode side was filled with solution, care being taken to avoid stirring up the silver iodide electrode. To accomplish this the solution was poured very slowly through a dropping funnel with a long stem, at the lower end of which was a small upward hook with a

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1) Bulletin of the Bureau Standards Vol. I, 34.

small opening . While the hydriodic acid was being added the funnel was gradually raised so that the liquid would be delivered just at the surface of the solution already in the apparatus. After this the other sections also were filled with the solution. Air bubbles were removed by means of a clean dry stirring rod as far as was possible. The anode silver spiral was fixed so as to occupy a position about 3 cm. above the stopcock. This left considerable solution above the electrode which stirred slowly during the run and prevented the exhaustion of the electrolyte in the immediate vicinity of the electrode. The whole apparatus was then placed in the thermostat to such a depth that the water and the solution in the tube were at the same level.

The connections were then made as shown in the diagram. The two coulometers were mounted on brass ring stands with glass stems, which prevented the leakage of the current. A voltmeter was connected across the switch, and a mili-ammeter and a rheostat were connected in series. The lead wires from the electrodes were protected from the water of the thermostat by rubber tubings. As a further precaution, a piece of rubber tubing was drawn over the bottom of the cathode part as far as the stopper, and was then brought out above the surface of water.

After the solution had come to the temperature of the thermostat, which was maintained at 25°C. the circuit was closed, and the rheostat, R , adjusted to give the proper current strength. The strength of the current, and the time of run varied with the concentration of the solution. In most cases the current used was about 30 mili-amperes.

As soon as the experiment was completed, the circuit was broken and the stop-cock closed. Then by introducing a clean, dry pipette into the apparatus through the small tubes at the top, samples of the three middle portions were obtained. These were transferred to 300 c.c. stoppered, tared Erlenmeyer flasks and weighed. After the removal of these portions the anode and cathode lead wires were disconnected, and the apparatus was taken out of the thermostat, dried on the outside, and the rubber tubes removed. The apparatus was then disconnected at d and e and the anode and cathode sections were stoppered with paraffined rubber stoppers. Next, the glass stop-cock in the anode section was opened and the anode portion thoroughly mixed by shaking. The contents of the cathode section were mixed in a similar manner. The two parts of the apparatus containing the electrode portions were then cleaned and dried on the outside and weighed to the nearest miligram. As much of the clear solution from the anode as possible was then transferred to a tared, stoppered, Erlenmeyer flask and weighed. The anode, silver spiral was washed and dried with alcohol. The anode chamber also was washed thoroughly and dried with alcohol. Then the anode section and the anode silver spiral were weighed. This weight subtracted from the weight of the anode section plus the solution gave the weight of the anode portion. The bulk of cathode portion was filtered through a tared Gooch crucible into a clean dry filter flask. The filtrate was immediately transferred into a tared Erlenmeyer flask and weighed and served as a sample for analysis. The residue from the cathode, which consisted of gray

metallic silver and silver iodide was left in the Gooch crucible and washed, dried, and weighed. The empty cathode section was also cleaned, dried and weighed. In order to obtain the weight of the total cathode portion the weight of the cathode section plus the weight of the silver and silver iodide in the Gooch crucible was subtracted from the weight of the cathode section before it was emptied.

All the solutions were analyzed by precipitating the iodide as silver iodide by a 0.03 normal silver nitrate solution. The hydriodic acid solution before precipitation was decolorized by a few drops of sodium sulphite and then diluted to 0.03 normal. Baxter[1] has shown that at this concentration of hydriodic acid and silver nitrate there is little danger of occlusion taking place during the precipitation.

Calculation of Results. - The calculation of the transference numbers given in Table I was made in the following manner. The mass of water in the electrode portion after electrolysis is obtained by subtracting from the weight of the portion the weight of hydriodic acid contained in it. The mass of the water is then multiplied by the ratio of hydriodic acid to water in the original solution, thus giving the mass of hydriodic acid in that amount of water before the electrolysis. To obtain the mass of hydriodic acid transferred, the mass of hydriodic acid present in the electrode portion before electrolysis is subtracted from that present after electrolysis in the case of cathode portion, and vice versa in the case of anode portion. By dividing the number of equivalents of hydriodic acid transferred by the number of equivalents

1) Baxter, Proc. Am. Acad. Arts and Sci. 41, 78 (1905)

of electricity passed through the solution the transference number of cation is obtained.

The results of the transference number measurements are given in Table I, the number at the top of each column being the number of the experiment. Concentrations are expressed in equivalents per 1000 g. of the solution.

Table I

Transference Number of Hydroidic acid at 25°, Experimental Data.

	1	2	3	4
Concentration	0.2N	0.16N	0.09N	0.06N
Wt. Ag. in coulometer A	1.3272	1.0084	0.6891	0.7239
" " " " C	1.3269	1.0076	0.6886	0.7239
" electrode portion A	197.313	203.471	192.580	202.811
" " " C	237.704	239.261	236.663	236.376
% HI in portion Mc	------	2.1064	1.1897	0.70501
% " " " M	2.6527	2.1060	1.1889	0.70452
% " " " Ma	2.6528	2.1070	1.1888	0.70392
% " " " A	2.0112	1.6317	0.84169	0.36619
% " " " C	3.1818	2.5085	1.4686	1.0233
Equivalents of electricity	0.012301	0.009344	0.0063859	0.006710
Grams of HI transferred (A)	1.2987	0.9859	0.6762	0.7076
" " " " (C)	1.2915	0.9839	0.6700	0.7035
Transference number of cation - - - - - - - - - - - - (A)	0.825	0.825	0.828	0.825
" " " " (C)	0.821	0.823	0.820	0.819

The values of the transference number contained in Table I are summarized in Table II

Table II

Transference Number of Hydriodic Acid at 25°

Concentration		Transference number: Calculated from anode portion	Transference number: Calculated from Cathode portion
0.20		0.825	0.821
0.16		0.825	0.823
0.09		0.828	0.820
0.06		0.825	0.819
	Mean	0.826	0.821
	A. D.	±0.001	±0.001

It appears from this table that in every case the transference number calculated from the change in concentration of the cathode portion is smaller than that calculated from the anode portion. Consequently in view of the many side reactions which can occur at the cathode, such as adsorption of the hydiodic acid by the silver iodide, and formation of the complex acid $HAg\ I_2$, either of which could cause a deceded change in the concentration of the cathode portion of the solution, the transference number calculated from the cathode side was discarded. Therefore, the value, 0.826, obtained from the concentration change in the anode portion was adopted as the transference number of the cation of hydriodic acid.

III. Conductance Measurement

The conductance measurements were made by the familiar Kohlraush

method, employing the pipette form of conductance cell in use in this laboratory[1] and the very accurate bridge wire described by Mr. J. E. Bell.[2] All of the glass apparatus which came in contact with the solutions the conductance of which was to be measured were cleaned carefully with warm chromic acid cleaning mixture and steamed for several hours before use.

The water used was conductivity water prepared in a special still and having a specific conductance of 0.6×10^{-6} at 25° C.

The hydriodic acid was prepared immediately before use by twice distilling the crude concentrated solution in the dark. The two successive distillations need not occupy more than half an hour, the distillate obtained being but very faint yellow and containing less than 0.01 per cent of free iodine. The acid so obtained was diluted to the desired concentration and called the stock solution, the more dilute solutions being made by weighing the required amount of the stock solution in a flask and diluting it with a weighed amount of conductivity water. The density at 25° of each solution was measured in order that its concentration by volume could be calculated. The concentration of the stock solution only was determined by precipitating the hydriodic acid as silver iodide, that of all other solutions being calculated from the concentration of the stock solution. Two stock solutions were made, having the concentrations of 0.2 normal and 0.02 normal, respectively, the former of these being used in the preparation of solutions stronger than 0.02 normal and the latter for those more dilute than 0.02 normal.

Immediately after the preparation of each solution its conductivity was determined the solutions remaining almost, if not quite, colorless during the measurement. After filling the

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1) Washburn and MacInnes, J. Am. Chem. Soc. 33, 1686-1713 (1911)
2) Washburn and Bell, J. Am. Chem. Soc. 35, 177-184 (1913).

conductance cell it was placed in the thermostat at $25^{\circ} \pm 0.01^{\circ}$ and left there a short time. The conductance was then measured twice at intervals of fifteen minutes apart in order to be sure that the solution had attained the temperature of the bath. The cell was then emptied and refilled with the solution and another measurement made. The various determinations of the conductance of the same solution agreed within less than 0.1 per cent. The conductance cell was one that has been in use in this laboratory for a number of years and has a cell constant of 0.6222. The cell was calibrated again by use of 0.1 normal potassium chloride just before being employed in this investigation, and the constant found to be unchanged.

The results of the conductance measurements are shown in Table III. Column I contains the concentration in equivalents per liter of the various solutions measured, and column II their respective equivalent conductances expressed in reprocal ohms. The figures in column IV are the equivalent conductances at the round concentrations shown in column III. They were obtained from the figures in column I and II by graphical interpolation. The conductances of solutions above 0.01 normal were read off a curve obtained by plotting the equivalent conductance, Λ, against the concentration, C. In order to arrive at the conductances at round concentrations below 0.01 normal and the equivalent conductance at zero concentration, it was found best to plot $\frac{1}{\Lambda}$ against $(C\Lambda)^n$ in the usual way, employing different values of n until a value was found that made the curve a straight line. In this way 0.40 was hit upon as the value of n. The equivalent

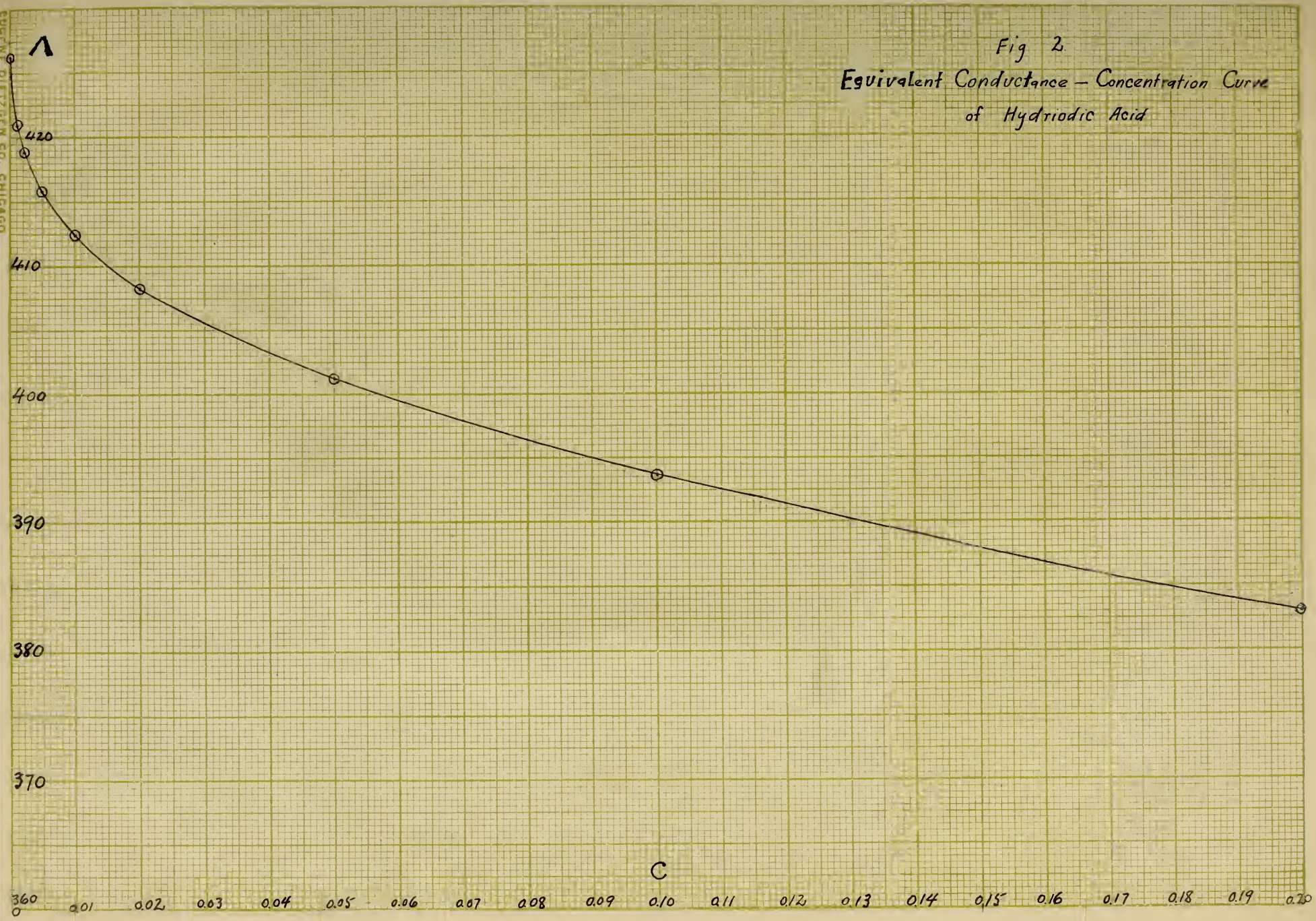

Fig 2.
Equivalent Conductance – Concentration Curve of Hydriodic Acid

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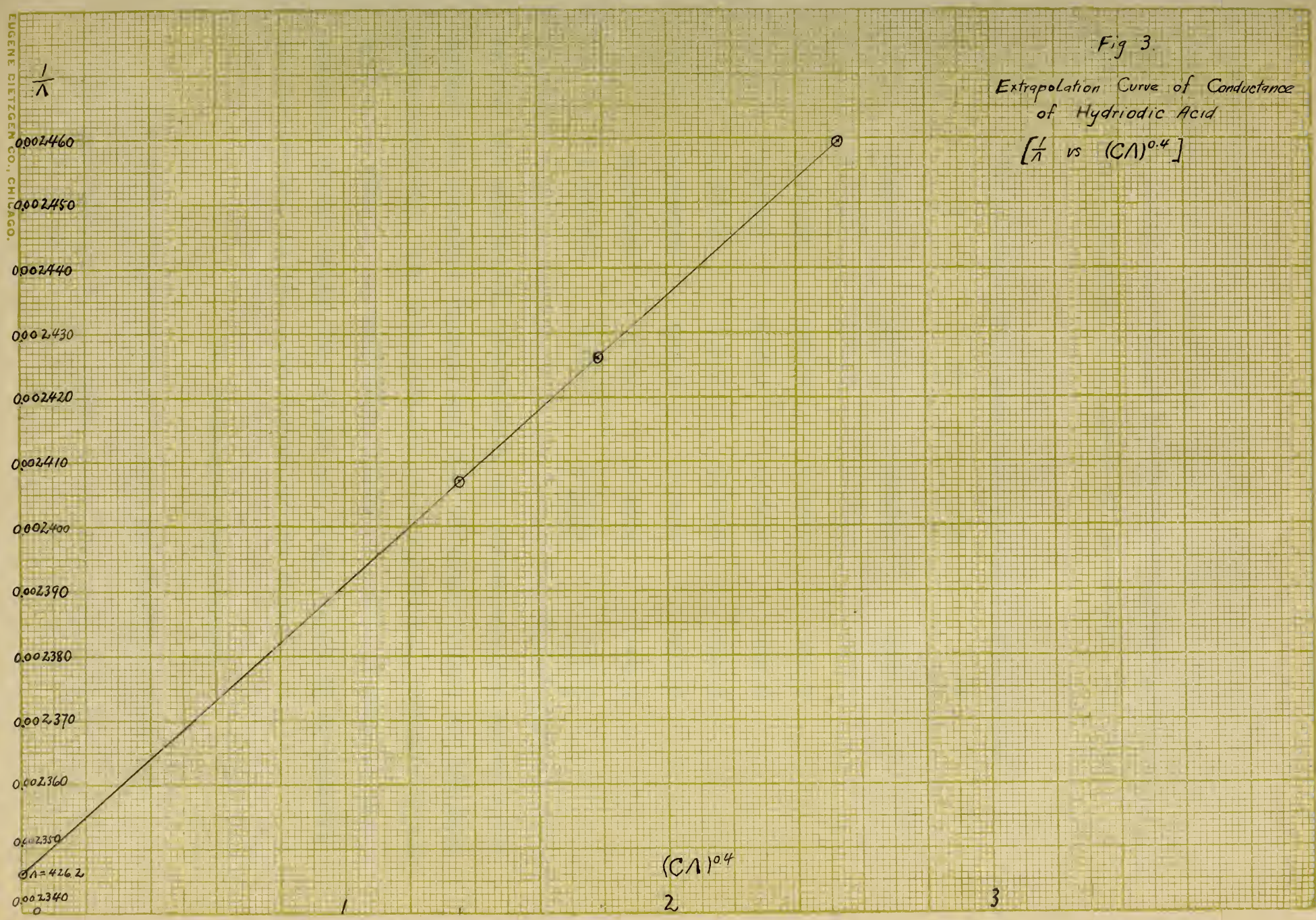
Fig 3.
Extrapolation Curve of Conductance of Hydriodic Acid
[1/Λ vs (CΛ)^0.4]
1/Λ
0.002460
0.002450
0.002440
0.002430
0.002420
0.002410
0.002400
0.002390
0.002380
0.002370
0.002360
0.002350
0.002340
Λ=426.2
(CΛ)^0.4
0
1
2
3
EUGENE DIETZGEN CO., CHICAGO.

conductance at zero concentration was found to be 426.2. The equivalent conductance of the iodide ion[1] may be taken to be 76.5 which makes that of the hydrogen ion appear practically 350, a result obtained by a number of observers. It will

Table III

Equivalent Conductance of Hydriodic Acid at 25°

I C	II Λ	III C	IV Λ
0.2217	381.2	0.2000	383.0
0.1124	391.8	0.1000	393.6
0.05435	400.4	0.0500	401.2
0.02507	406.6	0.0200	408.2
0.01036	412.2	0.0100	412.4
0.005144	415.5	0.0050	415.7
0.002152	417.5	0.0020	418.7
0 001009	418.6	0.0010	420.8
		0.0000	426.2

be noted that in column IV of the table the values finally accepted for the equivalent conductance at concentrations below 0.005 normal are somewhat higher than the measured values at the same concentrations. The interpolated values, however, probably represent more nearly the true conductance of the acid than do the measured values since the latter are complicated by an unknown correction due to the conductance of the water.

IV. Viscosity Measurement.

The viscosity of hydriodic acid was measured with the

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1) Bray and MacKay, J. Am. Chem. Soc., 32, 914, (1910).

apparatus of Mr. G. Y. Williams of this laboratory.[1] Through his kindness his precision quartz viscosimeter and entire apparatus were employed in this experiment, and with his effective aid the viscosities of two solutions of hydriodic acid of 0.1 and 0.2 normal concentrations were measured at 25°.

The solutions of hydriodic acid employed in this experiment were prepared in the same manner as those used in the conductance measurements. The density of each solution was simultaneously determined at 25°. The solutions were analyzed in the same way as before. At 25° the relative viocosity of 0.1963 normal solution of hydriodic acid was found to be 0.99542 and that of 0.09839 normal solution 0.99660. The value of viscoisty of a hydriodic acid solution at any intermediate concentration was obtained by linear interpolation.

V. The Degree of Ionization.

The calculation of the degree of inoization or dissociation of any electrolyte from conductance measurements depends upn a knowledge of the conductance of electrolyte when it is completely dissociated and of its conductance in the solution in which one desires to know the degree of dissociation. The latter value of the conductance is easily obtained by direct measurement, but that of the completely dissociated substance can be obtained only by a process of calculation. The usual method is to extrapolate a curve obtained by plotting $\frac{1}{\Lambda}$ against $(C\Lambda)^n$ to zero concentration. The use at higher concentrations of such a

1 Washburn and Williams, J. Am. Chem. Soc., 35, 747 (1913).

value of the conductance of the completely dissociated substance is open to criticism in all cases owing to the change of mobility of the ions with the change in viscosity of the solution. It is especially unreliable in the case of strong acids, for with them it is not only impossible to find a satisfactory expression relating the change in mobility of the hydrogen ion with changing viscosity of the solution, but also owing to some at present unknown cause, the equivalent conductance of the hydrogen ion appears to be quite different at zero concentration than at concnentrations above 0.002 normal,[1] as was noted in Chapter I.

These considerations have led to the development of a new method[2] of calculating the degree of dissociation of an acid, which involves no assumptions with regard to its conductance at zero concentration, nor to the change in equivalent conductance of the hydrogen ion with the concentration. The method is as follows.

The sum of the equivalent conductances of the ions of hydriodic acid, that is, the conductance of the acid if it were completely dissociated, is given by the relation

$$\Lambda_H + \Lambda_I = \frac{\Lambda_I}{T_a} , \qquad (1)$$

where T_a is the transference number of the anion and Λ_H and Λ_I are the equivalent conductances of the ions at the concentration under consideration. The equivalent conductanes of the ions will, however, not be the same in the solution as they are at zero concentration. The change of the equivalent conductance

1) Noyes et. al. loc. cit.
2) Washburn and Strachan, J. Am. Chem. Soc., 35, 681 (1913)

of the iodide ion (or that of any other ion but hydrogen or hydroxyl) however, is expressed by the relation[1].

$$\Lambda_I = \frac{\Lambda^\circ_I}{\left(\frac{\eta}{\eta_0}\right)}, \qquad (2)$$

where Λ°_I is the equivalent conductance of the iodide ion at zero concentration and $\left(\frac{\eta}{\eta_0}\right)$ the relative viscosity of the solution. Now since the degree of dissociation of any electrolyte is equal to the actual equivalent conductance of its solution, Λ_c, divided by the equivalent conductance of the completely dissociated substance, we may write for γ, the degree of dissociation of hydriodic acid. acid.

$$\gamma = \frac{\Lambda_c\left(\frac{\eta}{\eta_0}\right)T_q}{\Lambda^\circ_I} \qquad (3)$$

The values of γ, calculated by means of equation (3) are shown in column VI of Table IV. Columns I and II contain respectively the concentrations and equivalent conductances obtained from Table III. Column III contains relative viscosities obtained from the measured values by linear interpolation. Column IV contains the equivalent conductance of completely dissociated hydriodic acid at various concentrations calculated by use of equation (1) and column V the equivalent conductance of hydrogen ion obtained by subtracting from the values in column IV the equivalent conductance of the iodide ion as calculated by use of equation (2)

TABLE IV

Degree of Dissociation of Hydriodic Acid at 25°

I	II	III	IV	V	VI
C	Λ	$\left(\frac{\eta}{\eta_0}\right)$	$\Lambda_H + \Lambda_I = \frac{\Lambda^\circ_I}{T_q\left(\frac{\eta}{\eta_0}\right)}$	Λ_H	γ
0.20	383.0	0.9954	441.7	364.9	86.70

1) Washburn, J. Am. Chem. Soc. 33, 1469 (1911). In the present discussion m in Washburn's formula may be taken as unity without introducing any appreciable error.

Table IV Cont.)

I	II	III	IV	V	VI
C	Λ	$\left(\frac{\eta}{\eta_0}\right)$	$\Lambda_I+\Lambda_H$	Λ_H	γ
0.10	393.6	0.9966	440.8	364.1	89.30
0.05	401.2	0.9980	440.5	363.9	91.07
0.02	408.2	0.9992	440.0	363.4	92.77
0.01	412.4	1.0000	439.7	363.2	93.80
0.005	415.7	1.0000	439.7	363.2	94.55
0.002	418.7	1.0000	439.7	363.2	
0.001	420.8	1.0000	439.7	363.2	
0.000	426.20	1.0000	439.7	363.2	

It appears from examination of the table that the degree of dissociation of hydriodic acid is less than that commonly ascribed to it. Moreover, the value obtained for the equivalent conductance of hydrogen ion is very close to 365, a value obtained by a number of observers.

VI. Summary and Conclusions.

1. The transference number, the equivalent conductances, and the relative viscosities of solutions of hydriodic acid at 25° have been measured.

2. The value 0.826 was adopted as the transference number of cation of hydriodic acid at 25°.

3. The value for the transference number of cation by analysis of cathode portion of the solution was always lower than that calculated from the analysis of the anode portion. That is very likely due to adsorption of hydriodic acid by the

silver iodide and to other side reactions of the silver iodide cathode. It is, therefore, desirable that the transference number of hydriodic acid be measured by some means which is not open to this criticism.

4. By extrapolation of conductance data the equivalent conductance of hydriodic acid at zero concentration was found to be 426.2 which indicates that the equivalent conductance of the hydrogen ion at this concentration is 350 if that of iodide ion is 76.5

5. The equivalent conductance of completely dissociated hydriodic acid at concentrations higher than 0.001 normal is approximately 440, making the equivalent conductance of the hydrogen ion 364.

6. The relative viscosity of hydriodic acid is less than unity.

7. The degree of dissociation of hydriodic acid calculated by the method described in Chapter V appears to be considerably smaller than the commonly accepted values.

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